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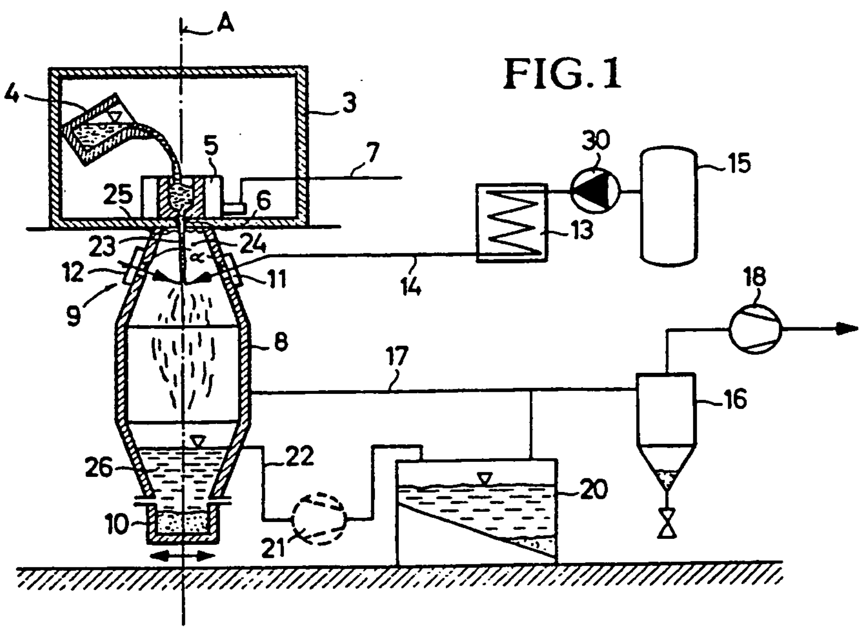
(54) **Process and apparatus for producing powder from a melt by atomization**

(57) For producing powders of the finest particle size, high purity and uniformity, low-boiling liquefied gas is sprayed through the atomizing device 9 under a pressure of 50 to 700 bar on the melt stream 23 entering the spray diffusion chamber 8 through several nozzles 11, 12,......) which can be oriented in their mountings towards the melt stream. A heat exchanger 13 is connected between high pressure pump 30 and the nozzles for cooling the liquid gas.

FIG.1



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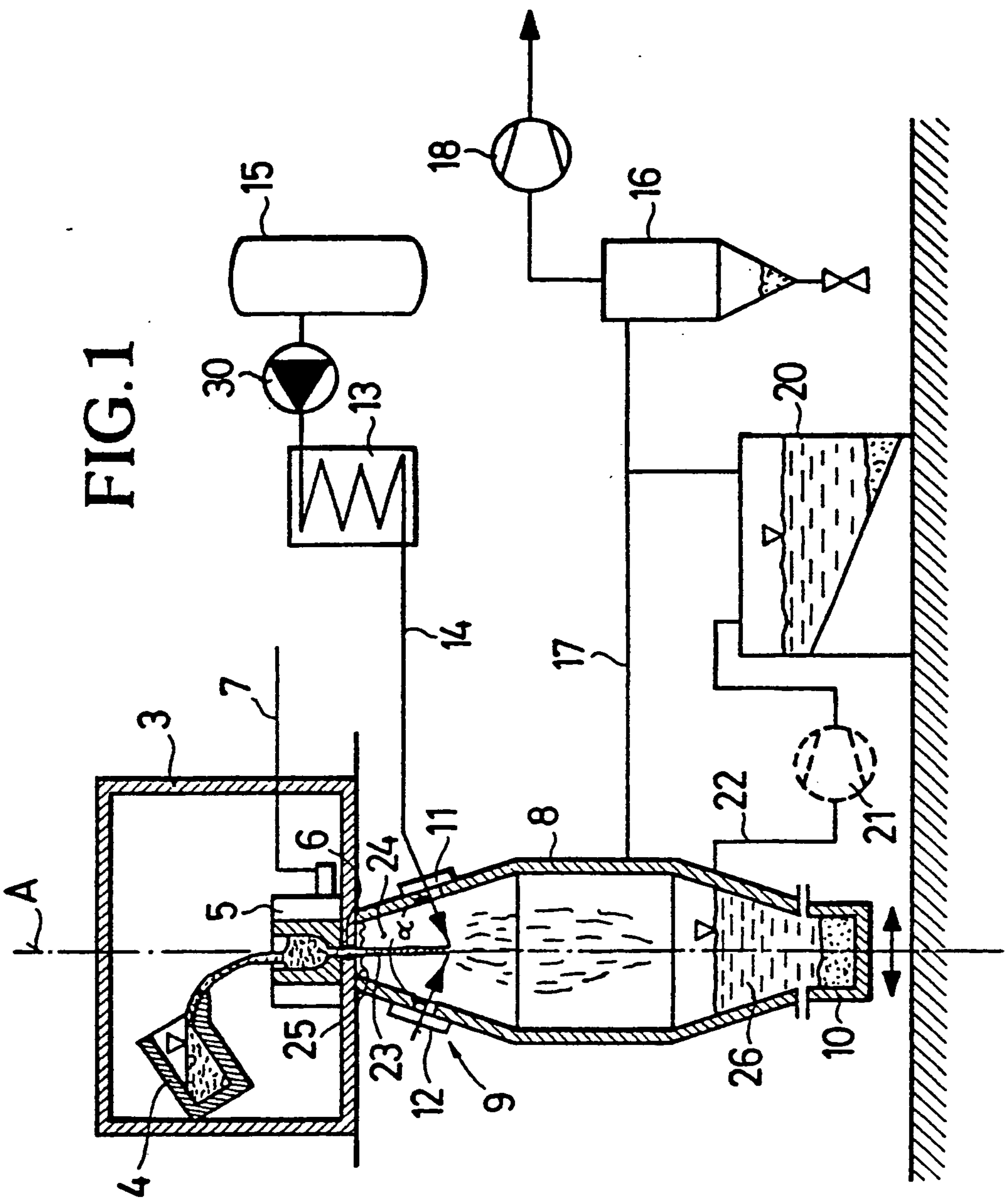

FIG.1

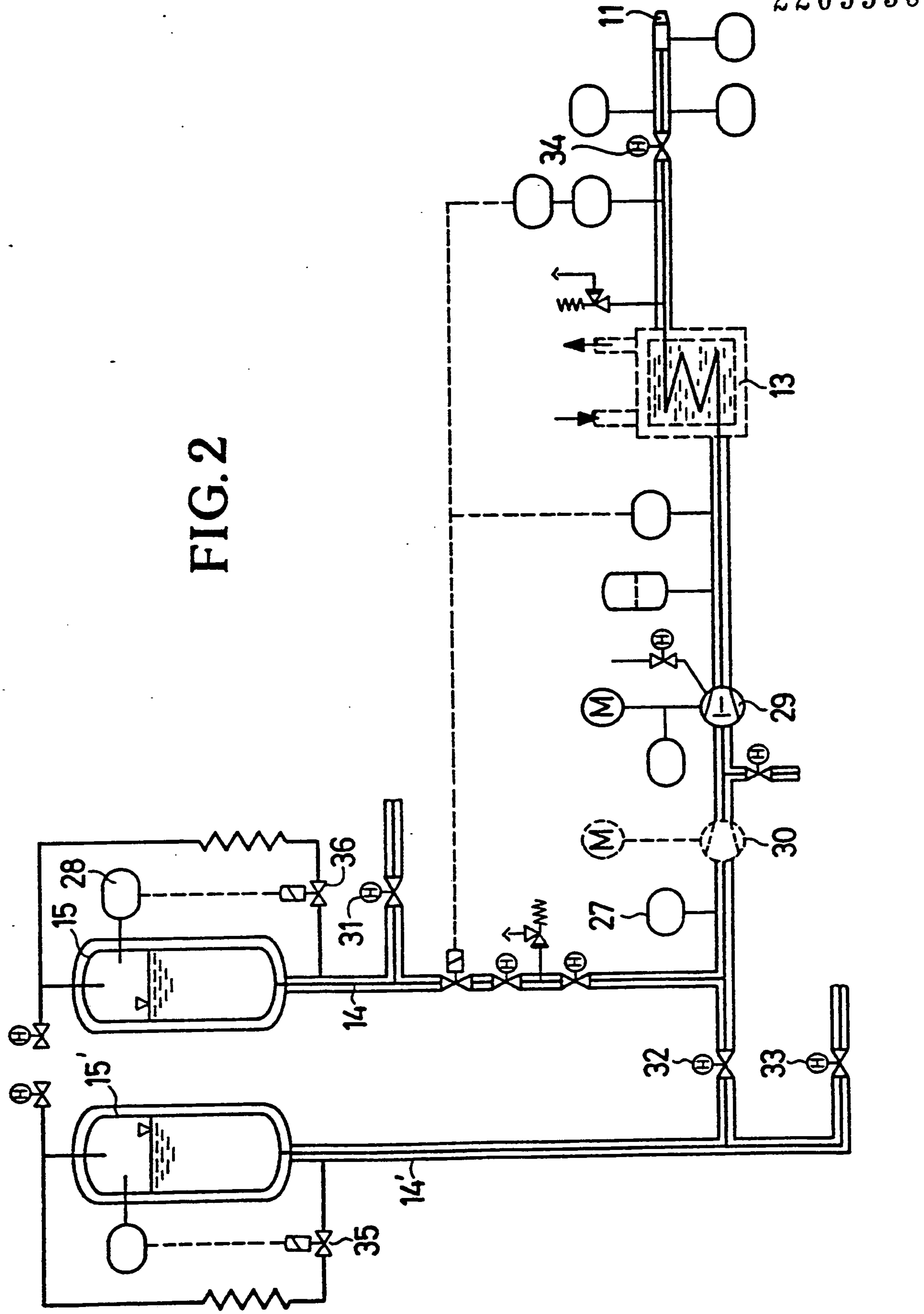

FIG.2

PROCESS AND APPARATUS FOR PRODUCING POWDER FROM A MELT

The invention relates to a process and apparatus for producing powder from a melt.

It is known that powders, for example metal powders, can be produced from a melt by spray diffusion (DOS 30 34 677). In this process, a jet of the melt is dispersed into fine droplets by means of a gas or a liquid, such as water or oil at high relative velocity to that of the melt, which droplets then solidify in a subsequent cooling tract.

A disadvantageous or restrictive feature of this process is the fact that gas-dispersed powders only have a coarse relative particle size, typically between 40 and 150 µm. Even in the case of ultrasound-dispersed powders, only a gradual reduction of the mean particle size can be achieved. To this must be added the poor cooling effect of the gas in the following cooling tract, especially below, say, 600°C which, in the case of metal alloys, such as aluminium-base metals, can lead to undesirable segretation during powder production.

In the case of water or oil-dispersed powders, smaller mean particle sizes can be obtained than is possible in the case of dispersion with gases. However, the powders obtained carry, at least on the surface of the particles, undesirable reaction products such as oxides, hydrides, carbides, etc. (in the case of dispersion by gases this can be avoided by the use of inert gases).

Additional difficulties can arise in the case of water or oil-dispersed powders by the necessity of having to separate the powders from the dispersing

medium.

Furthermore a process for producing fine metal powders is also known, in which a jet of molten metal is introduced into the orifice of a vessel by the action of a gas flowing at supersonic velocities, wherein the ratio of the gas pressure in the vicinity of the orifice outside the vessel and of the gas pressure inside the vessel is adjusted to a value greater than 5, and wherein the gas streaming into the vessel prior to entering the vessel is at a temperature in the range of 0.7 to 1.5 times the solidification temperature of the metal (in °K), wherein the melt stream is first divided into filaments which, in the region of the negative pressure, are converted into droplets in the hot gas and wherein these droplets solidify into sphereical metal powders (DPS 33 11 343). In this process, the melt is brought into contact with the gas at that point of the vessel orifice at which the gas pressure has fallen to less than 60% of the pressure in front of the orifice. This previously known process makes it possible to produce very finely divided powders with particle diameters of less than 40 µm, with a relatively narrow particle size distribution and with a relatively low energy consumption.

An object of the present invention is to provide a process and an apparatus suitable for producing gas-dispersed powders of extreme uniformity and purity and of still finer particle size.

This is achieved according to the invention by a process for producing powder from a melt, which comprises melting a substance to be converted to powder form, directing a jet stream of the melt downwardly into a spray diffusion chamber, dispersing the jet stream of melt by spraying a low-boiling liquified gas under pressure onto the melt stream entering the

diffusion chamber, cooling the dispersed particles of melt during their falling movement through the diffusion chamber, and collecting the resulting powder.

The liquefied gas is preferably under a pressure of more than 50 bar and is injected through one or more nozzles directed onto the jet of melt, so that the liquid gas jet issuing from one or more of the nozzles and the stream of melt flowing perpedicularly downward from the melting chamber form an angle which is smaller than a right angle.

Advantageously, liquid nitrogen or liquid argon at a pressure of 50 to 700 bar is sprayed on the stream of melt. The flow rate of the low-boiling, liquefied gas is expediently so adjusted, that the amount of heat which can be extracted from the melt by the liquid gas is more than twice, preferably 8 to 10 times, the amount of heat which must be extracted from the melt in order to cool the latter to ambient temperature.

It may be expedient to cause the melt to enter the spray diffusion chamber under supra-atmospheric pressure in order to ensure that the melt stream is endowed with a precisely definable quality, that is to say, velocity, shape and uniformity.

Depending on the selected alloy components, it may be desirable to produce the melt under vacuum and to feed it to the atomizer without contact with atmospheric oxygen. It can be similarly desirable to pre-cool the spray diffusion chamber and to cool the atomizing medium, in order to effect that the gas jets issuing from the nozzles and directed on the melt stream remain parallel as far as possible.

The invention also provides apparatus for carrying out the aforedescribed process and which comprises a melting chamber for preparing the melt, a spray diffusion chamber arranged below the melting

chamber, means for directing a stream of melt into an upper region of the diffusion chamber, means including one or more nozzles arranged in the upper region of the diffusion chamber for spraying a low-boiling liquefied gas into the upper region of the chamber to act on and disperse the melt stream, means including a high pressure pump and a pressure line for supplying the gas to the nozzles, and means for collecting the resulting powder from the lower regions of the diffusion chamber, wherein a heat exchanger is connected into the pressure line between the high pressure pump and the nozzles for cooling the liquid gas to or close to its boiling temperature at normal pressure by means of a low-boiling liquefied gas, such as liquid nitrogen.

In order to exclude undesirable reactions between the melt entering the cooling chamber and the ambient air, a protective-gas atmosphere can be advantageously established in the spray diffusion chamber prior to atomization.

In order to exclude an undesirable pollution of the spray diffusion chamber even prior to the atomizing process, the spray diffusion chamber can be sealed off from the melting chamber by a membrane or a valve which only melts at the instant when the melt impacts on it.

The nozzles for the low-boiling liquefied gas are preferably fastened to pivotably mounted retaining means on the wall of the diffusion chamber so that the position of the nozzles can be varied from the outside with great precision horizontally and vertically in the range between 30° and 90°.

It is preferred to mount the nozzles, which are preferably constructed as flat or round-jet nozzles, so as to be longitudinally displaceable in their retaining means so that their distance relative to the melt stream is reducible in such a manner as to

exclude a broadening of the liquid gas jet.

The invention can be practised in various ways; a particular embodiment will now be described by way of example, reference being made to the accompanying generally diagrammatic drawings in which:-

Fig. 1 is a diagrammatic illustration of apparatus in accordance with the invention for producing powders from a melt,

Fig. 2 is a circuit diagram showing the supply of a low-boiling liquefied gas to the atomizing device of the apparatus according to Figure 1.

Referring to Figure 1, the apparatus shown comprises a melting chamber 3. Arranged in the melting chamber 3 is a melting device 4 and a heated crucible 5 with an outlet opening 6 and current input leads 7. A spray diffusion chamber 8 is arranged below the melting chamber 3 and is thermally insulated therefrom. The upper region of the spray diffusion chamber 8 defines a jet dispersion space 24 and is sealed from the crucible 5 by a diaphragm 25 or a valve. Fitted to the wall of the chamber 8 in the upper region is an atomizing device 9 including nozzles 11, 12 for injecting a low-boiling liquefied gas under pressure against a stream of melt 23, issuing from the outlet orifice of the crucible 5, at an angle α relative thereto which is less than a right angle. The nozzles 11, 12 are preferably fastened to retaining means pivotably mounted and exteriorly accessible so that their angle can be varied with precision horizontally and vertically in a range of 30° to 90°. The nozzles 11, 12 may also be longitudinally displaceable so that their distance from the melt stream 23 is variable for negligible broadening of the jet stream.

A gas tank 15 holds a supply of the low-boiling liquefied gas which is fed by a high pressure pump 30 (50-700 bar) to a heat exchanger 13 where it is

cooled to or close to its boiling temperature at normal pressure by means of a low boiling liquefied gas, such as nitrogen, and thence by a pressure line 14 to the nozzles 11, 12.

Gas is extracted from the chamber 8 through an extractor line 17 and a cyclone 16 by a fan 18. Lastly, a powder-collector 20 with a suction pump 21 is connected into a suction line 22 from the chamber 8.

As Figure 2 shows, instead of a single gas tank 15 there may be provided two gas tanks 15, 15', containing liquefied nitrogen or liquefied argon. The pressure lines 14 or 14' can be connected with a number of pressure/temperature and flow-rate monitors 27, 28, so that before and during the process the gas feed to the nozzles 11 or 12 can be accurately measured and regulated through high-pressure pumps 29, 30 or shut-off elements 31 to 36, respectively.

Four flat-stream or round-stream (full-flow) nozzles 11, 12,.... arranged perpendicularly relative to each other and having a nozzle aperture of 0.5 to 2 mm are so adjusted in their mountings that, firstly, they form an angle of between 30° and 90° to the vertical and, secondly, are oriented to the axis A of the spray diffusion chamber 8, in which the outlet aperture 6 of the crucible 5 is also located and, thirdly, every two of the flat streams 11, 12,.... intersect in a line (in case of full-flow nozzles at a spot), at least, at the smallest possible distance from the axis of the spray diffusion chamber 8. This arrangement prevents the jet of melt 23 from missing the flat streams as they fall.

An example of the process for producing an aluminium alloy powder will now be described. The aluminium alloy is melted in the melting chamber 3, and poured into the crucible 5 which has an outlet nozzle (2 to 8 mm diameter) located at the bottom and closed

by a packing rod. The melting chamber 3 is located above the spray diffusion chamber 8 and is thermally insulated from the latter. The jet dispersion space 24 in the diffusion chamber 8 is closed by a membrane 25.

The jet dispersion space 24 is cooled by the injection of low-boiling liquefied argon gas until liquid argon settles out on the bottom of the jet dispersion space 24 and provides an inert atmosphere.

During the spray dispersion, after withdrawal of the packing rod (not shown in detail), the melt is formed by the outlet opening 6 into a fine, uniform and stably-flowing jet 23, which impacts on the membrane 25, melts the latter and then falls into the dispersion space 24. The dispersion rate is approximately 4 kg melt/min; to this end, approximately 300 l/min argon are used. The argon is compressed to approximately 250 bar and is cooled in front of the outlet aperture below the boiling temperature at normal pressure.

The gas evaporating during the dispersion process is evacuated and is separated in one or several cyclones 16 from entrained fine powder particles (0.5 - 10 um). After the dispersion and the settling out of the particles the liquid gas can be drawn off through the suction line 22 and the powder can be retrieved from the powder collector 20, the collector vessel 10, and the cyclone 16.

When dispersing melts of iron alloys, four flat-jet nozzles with a nozzle aperture of 0.5 to 2 mm and arranged perpendiculalry relative to each other are employed, in such a manner that, firstly, they form an angle of 90° to 30° to the vertical and, secondly, that they are oriented towards the axis of the spray dispersion chamber 8 in which the outlet opening 6 of crucible 5 is also located and, thirdly, at least two of the flat jets each intersect in a line, said jets being at a distance as small as possible from the axis

of the spray dispersion chamber 8.

The jet dispersion space 24 is cooled by the injection of low-boiling liquefied nitrogen until liquid nitrogen settles out on the bottom of the jet dispersion space 24. The nitrogen evaporating during cooling of the apparatus displaces the air from the jet dispersion space 24 and provides a protective atmosphere.

The iron alloy is melted down and poured into the (pre-heated) crucible 5, which has an outlet opening 6 (2 to 6 mm diameter) at the bottom. The crucible 5 is located above the spray dispersion chamber 8 and is thermally insulated from the latter. The jet dispersion space 24 is closed by a membrane 25.

In the outlet opening 6 the melt is formed into a fine, uniform and stably flowing jet 23, which impacts on the membrane 25 and melts the latter and then falls into the jet dispersion space 24. The jet dispersion rate is approximately 8 kg . melt/min; for this purpose, approximately 300 l/min liquid nitrogen are used. The nitrogen is compressed to approximately 600 bar and is cooled before the nozzles 11, 12,.... to a temperature below, equal to or close to its boiling temperature at normal pressure.

The gas evaporating during atomization is evacuated and is separated in one or more cyclones from entrained fine powder particles (0.5 - 10 µm), in which operation the powder particles can be classified at the same time.

At the bottom of the jet dispersion unit a sump 26 is formed of liquid gas and coarser particles (typically larger than 10 µm). After the jet dispersion and the settling out of the particles the liquid gas can be drained off and the powder removed.

CLAIMS

1. A process for producing powder from a melt, which comprises melting a substance to be converted to powder form, directing a jet stream of the melt downwardly into a spray diffusion chamber, dispersing the jet stream of melt by spraying a low-boiling liquified gas under pressure onto the melt stream entering the diffusion chamber, cooling the dispersed particles of melt during their falling movement through the diffusion chamber, and collecting the resulting powder.

2. A process according to Claim 1, wherein the low-boiling liquefied gas in injected under a pressure of more than 50 bar through one or more nozzles oriented towards the melt stream such that the liquid gas stream issuing one or more of the nozzles makes an angle of less than a right angle with melt stream flowing perpendicularly downwards from melting chamber.

3. A process according to Claim 1 or 2, wherein the low-boiling liquefied gas is liquid nitrogen or liquid argon and is sprayed on the melt stream at a pressure of 50 to 700 bar.

4. A process according to any one of the preceding claims, wherein the flow rate of the low-boiling liquefied gas is so adjusted that the amount of heat which can be extracted from the melt by the liquid gas is more than twice, preferably 8 to 10 times, the amount of heat which has to be extracted from the melt in order to cool it to ambient temperature.

5. A process according to any one or several of the preceding claims, wherein the melt enters the spray diffusion chamber at supra-atmospheric pressure.

6. A process according to any of the preceding claims, wherein the melt is fused under vacuum or an inert gas and is fed directly to the atomizing process without admission of air.

7. A process according to any of the preceding claims, wherein the spray diffusion chamber is pre-cooled.

8. A process according to any one of the preceding claims, wherein an inert gas atmosphere is provided prior to dispersion in the spray diffusion chamber.

9. Apparatus for producing powder from a melt by the process according to Claim 1, comprising a melting chamber for preparing the melt, a spray diffusion chamber arranged below the melting chamber, means for directing a stream of melt into an upper region of the diffusion chamber, means including one or more nozzles arranged in the upper region of the diffusion chamber for spraying a low-boiling liquefied gas into the upper region of the chamber to act on and disperse the melt stream, means including a high pressure pump and a pressure line for supplying the gas to the nozzles, and means for collecting the resulting powder from the lower regions of the diffusion chamber, wherein a heat exchanger is connected into the pressure line between the high pressure pump and the nozzles for cooling the liquid gas to or close to its boiling temperature at normal pressure by means of a low-boiling liquefied gas, such as liquid nitrogen.

10. Apparatus according to Claim 9, wherein the spray diffusion chamber is sealed off from the melting chamber by a membrane or a valve which melts down only in the instant when the melt stream impacts on it.

11. Apparatus according to Claim 9 or 10, wherein the nozzles spraying for the low-boiling liquefied gas

are fastened to retaining means pivotably mounted in the wall of an atomizing device accessible from the exterior of the spray diffusion chamber so that the position of the nozzles can be varied from the outside with high precision horizontally and vertically in a range between 30° and 90°.

12. Apparatus according to any one of Claims 9 to 11, wherein the nozzles are flat stream nozzles are longitudinally displaceably mounted and their distance from the melt stream is reducible in such a manner that broadening of the jet of liquid gas is negligible.

13. A process for producing powder from a melt, according to Claim 1 and substantially as hereinbefore described.

14. Apparatus for producing powder from a melt, substantially as hereinbefore described with reference to the accompanying drawings.

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